



Hydrogen generation by catalytic partial dehydrogenation of low-sulfur fractions produced from kerosene Jet A-1

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ABSTRACT

Fractionation of kerosene by rectification leads to fuel fractions characterized by different proportions of iso-paraffins, *n*-paraffins, cyclic, dicyclic, aromatics and diaromatics. Fractions of progressively higher boiling point range are characterized by progressively higher average carbon chain length, as well as increasing content of sulfur containing molecules. Three fractions, corresponding to 5, 14 and 32 wt.% of the original kerosene and having boiling point ranges 140–150 °C; 150–160 °C; 160–170 °C, respectively, containing predominantly C₈–C₉, C₉–C₁₀ and C₁₀, respectively, and 4, 12 and 24 ppm sulfur, respectively, were fed to a partial dehydrogenation reactor using a Pt–Sn/BaO–γ–Al₂O₃ catalyst. A hydrogen productivity increase up to a factor 3 for the low-boiling point fraction compared to the original kerosene is observed. The catalyst durability is significantly increased due to lower sulfur and coke deposition on the catalyst for the low boiling point fraction. The fractionation of kerosene Jet A-1 fuel by rectification is a promising approach for the enhancement of durable hydrogen generation via partial dehydrogenation.

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1. Introduction

Aircraft traffic gives a quantifiable contribution to the greenhouse effect emissions and other pollutants and about 25% of these are produced during landing/take-off operations when the auxiliary power unit (APU) supplies the power for on-land operations like cabin environment control, illumination or turbine start-up [1–4]. These emissions may be reduced by substituting the standard APU of commercial airplanes to a fuel cell based APU, fed with hydrogen generated from desulfurized jet fuel (kerosene Jet A-1) by partial dehydrogenation [5,6]. Desulfurization, however, increases the cost of kerosene compared with that of standard jet fuels and the supply cannot be assured in all areas of the world due to different legislations and environmental policies. An easier and practical alternative to the hydrosulfurization of kerosene Jet A-1 is its thermal fractionation by rectification. This process, comprises successive distillations for the separation of components with similar boiling temperatures, is very well-known and is extensively used

in petrochemical, chemical, pharmaceutical and food industries. In distillation generation of a two phase system is followed by mass transfer across the interface and separation of the two phases [7–10]. Since the sulfur-containing components of kerosene are usually benzothiophenes and derivatives that occur in the higher boiling range fraction of kerosene [11–12], this process can be used to lower the sulfur content by collecting the kerosene head product of the rectification, which has a lower boiling range. The remaining sulfur-enriched kerosene fraction as well as the dehydrogenated fractions of the kerosene fuel can be fed to the aircraft turbines.

Partial dehydrogenation (PDh) of hydrocarbon blends like kerosene (but also gasoline and diesel) has recently emerged as an innovative means of producing hydrogen for on-board electricity generation by feeding the hydrogen to a proton exchange membrane fuel cell (PEMFC). Alumina-supported Pt–Sn reforming catalysts have been investigated for their effectiveness for PDh using surrogate mixtures of kerosene [13–17]. More recently, modified Pt–Sn catalysts have been developed for PDh of desulfurized kerosene and ultra-low sulfur kerosene, ULSK, since the Pt–Sn catalyst is sensitive to sulfur poisoning. Various modification on the catalyst formulation and treatments have been carried out to improve the catalytic activity and the stability, including

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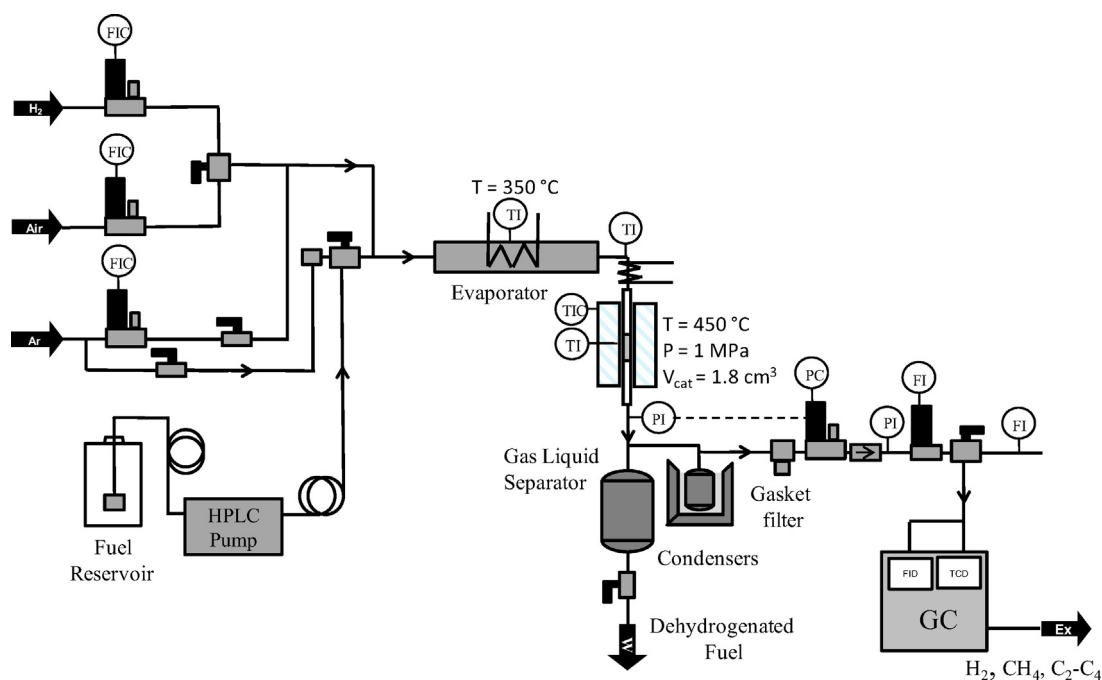


Fig. 1. Catalytic partial dehydrogenation test rig used for PDh of rectified and native Jet A-1.

incorporation of alkali metal ions [16,18], modification of the support [19,20] or modification of the catalyst active phase [21]. Particularly effective, in terms of performance enhancement of Pt-Sn based catalyst, seems to be the presence of barium oxide in the alumina support [19]. As discussed above, rectification is a practical method of decreasing the sulfur content and enriching the cut with light hydrocarbons, and combining the use of one of these enhanced catalysts with the rectification process is a promising opportunity to increase the efficiency of PDh and avoid the need for special low sulfur kerosene feedstock.

We describe here the effect on the PDh of kerosene of using rectified Jet A-1 fractions, using a multimetallic catalyst supported on γ - Al_2O_3 (1% Pt, 1% Sn, 3% BaO w/w). In order to determine the effectiveness of the process the results are compared to the PDh reaction performed on the original Jet A-1 fuel from which the fraction where obtained.

2. Materials and methods

The fractionation of Jet A-1 containing 228 ppm S was carried out in a laboratory scale Vigreux rectification column. Kerosene fractions were prepared for PDh by loading the raw kerosene in the boiler and taking the 5%, 14% and 32 wt.% as the rectification head product, taking each fraction from a new raw charge. The hydrocarbon components of kerosene and rectified kerosene were detected using a gas chromatography (GC) system with a flame ionization (FI) detector (Shimadzu GC-2010). Different components arrive in different time intervals at the detector and the integral over the measured detector signal in this time interval provides a measure for the total carbon mass of each component. This value was divided by the integrated signal of the detector over the total measuring time to give the mass fraction of each component. The components were identified with the help of gas chromatography/mass spectroscopy (GC-MS) (Thermo T230L Trace DSQ Turbo 250L sec 230) that provided around 300 component peaks of which 70 to 95% of the overall peak area were identified depending on the kerosene fraction analyzed. The compounds are classified here according to their chemical structure: iso-paraffins and n-paraffins, and cyclic, dicyclic, aromatic and diaromatic hydrocarbons. The chemical com-

position was also determined for spent fractions recovered after six hours of PDh reaction. Differences in the chromatograms of the kerosene fractions pre- and post-PDh reaction provide information on the dehydrogenation process for the component classes. The sulfur content of the fractions was analyzed using an elemental analyzer (*AnalytikJena/multi EA 5000*). The three fractions and the native Jet-A1 reference were used as feeds for the PDh reaction. The reaction was carried out in a stainless steel fixed-bed tubular reactor. Before reaction, the catalyst was activated for 5 h in a flow of synthetic air at 500 °C and reduced at atmospheric pressure with a H_2/Ar flow (4:6 v/v) at 350 °C for 2 h. A vapor flow of kerosene fraction of 25 g hr^{-1} with a 7% vol. (3.78 ml/min) of H_2 (to simulate a recycle), was fed with a volumetric pump to the reactor containing pelletized catalyst ($m=0.9$ g.; $d=0.85$ –1 mm, bed volume = 1.8 cm^3) at $T=450$ °C and $P=1$ MPa. The catalyst was synthesized by Johnson Matthey by impregnation of Pt and Sn metal precursors on a porous support of γ -alumina doped with 3 wt.% BaO [16]. The kerosene (or its fractions) vapor flow results in a contact time close to $t=2$ s (calculated at STP). After condensing the dehydrogenated kerosene fraction at room temperature using

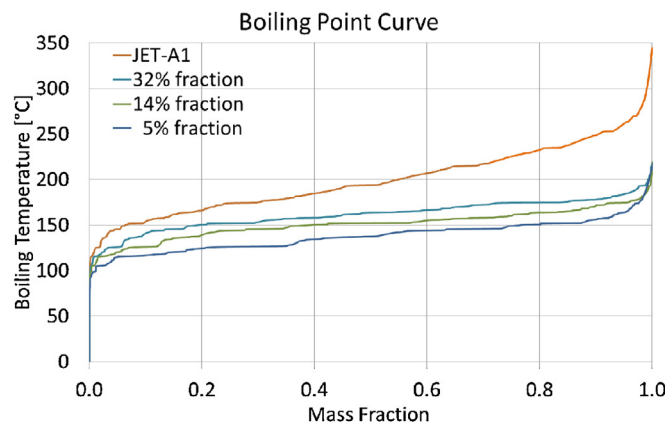


Fig. 2. Boiling point curves for Jet A-1 kerosene and three rectified fractions.

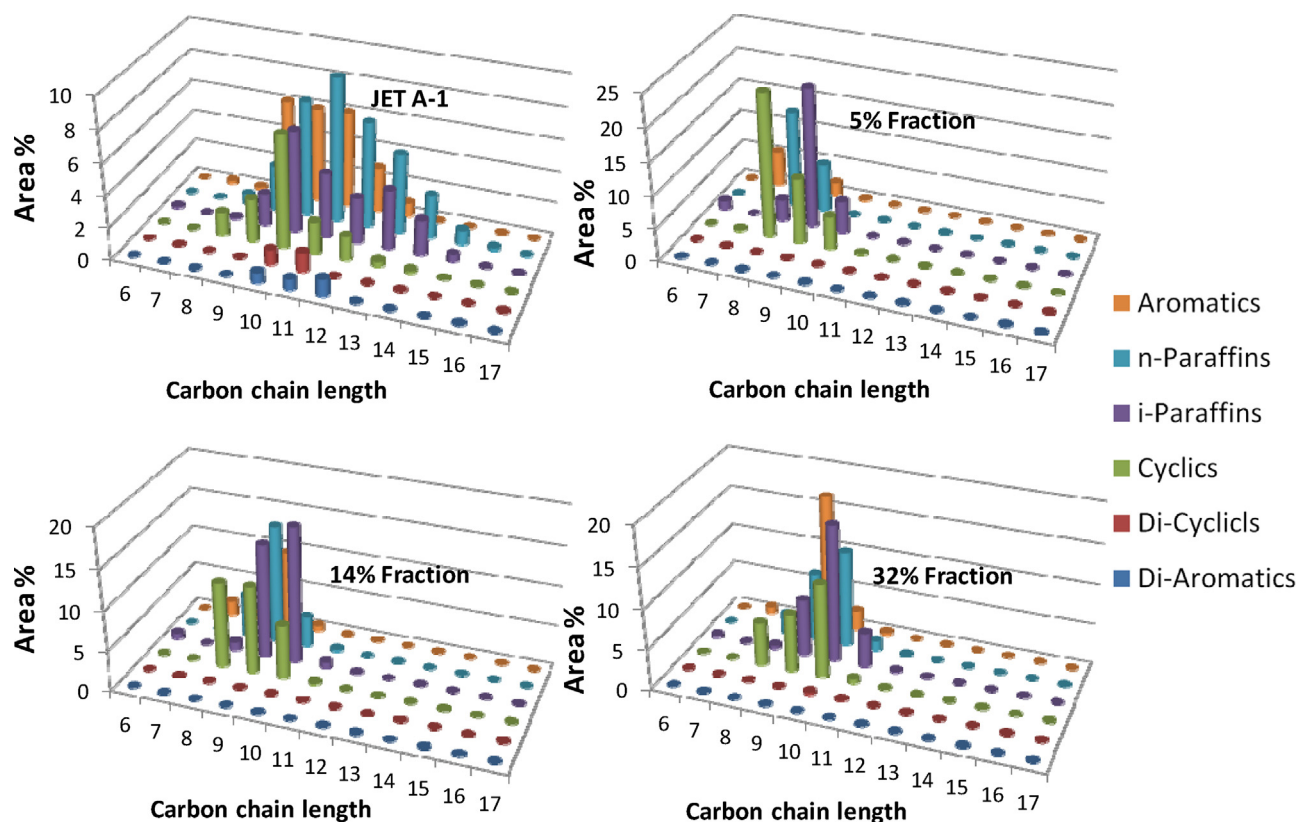


Fig. 3. Chemical distribution of kerosene Jet A-1 and its fractions.

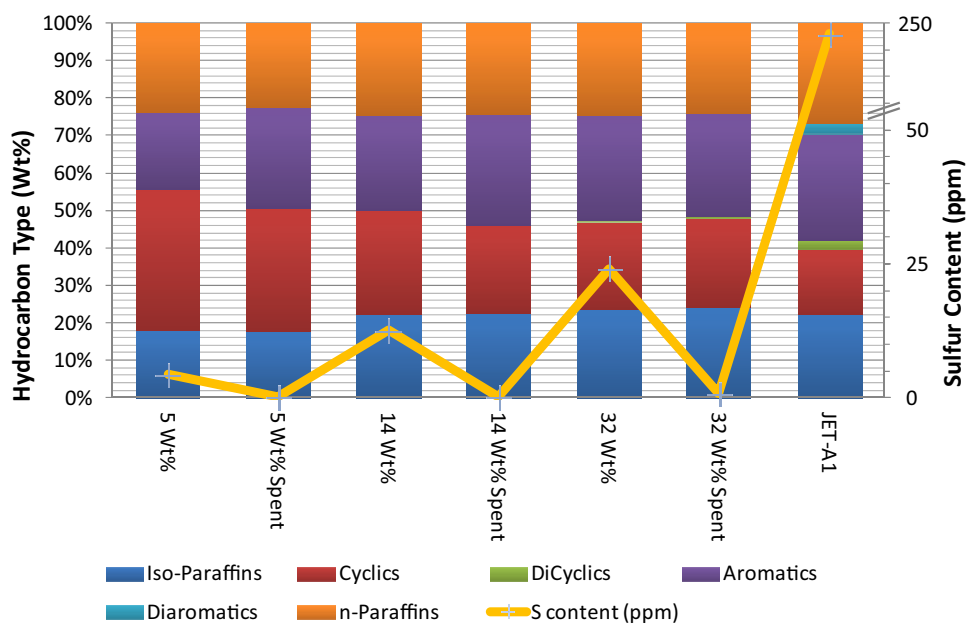


Fig. 4. Composition of Jet A-1 and its fractions in comparison to the compositions of the fractions after reaction.

a double condenser, the rate of hydrogen production ($\text{NL}_{\text{H}_2} \text{h}^{-1} \text{kg}_{\text{cat}}^{-1}$) was calculated from the measurement of the gas out-flow with a digital mass flow meter, subtracting the hydrogen recycle flow and the gas volume flow displaced by the condensed dehydrogenated kerosene fraction. Hydrogen purity was analyzed with a gas chromatograph Agilent 7890A with a dual column HP-PLOT molecular sieve 5A with thermal-conductivity (TC) detector to analyze the hydrogen and HP-PLOT/Q with an FI detector to analyze

the gaseous light hydrocarbons produced. A scheme of the reactor is shown in Fig. 1. Thermogravimetric and Differential Thermal Analyses (TG/DTA) were performed from ambient temperature till 800°C on used catalysts held in alumina crucibles with a Nietzsche STA409PC system, using a heating ramp of $10^\circ\text{C min}^{-1}$ in a synthetic air flow of 50 ml min^{-1} . Carbon, hydrogen and sulfur contents on spent catalysts were determined using a Thermo FLASH EA 1112 automated analyzer.

3. Results and discussion

3.1. Jet A-1 rectification

The three different kerosene fractions were prepared for the partial dehydrogenation reaction by rectification of a commercial Jet A-1. The kerosene fractions obtained comprise the 5%, 14% and 32% w/w of the head cut of the original Jet A-1. The experimental boiling point curves for the fractions and the original kerosene are reported in Fig. 2. The range of boiling temperature decreases with lower weight fraction. The compounds contained in the Jet A-1 and its fractions have been classified according to their carbon chain length and the chemical structure: iso-paraffins and n-paraffins; cyclic, dicyclic, aromatic and diaromatic hydrocarbons. The composition calculated from GC analysis is represented schematically in Fig. 3. Jet A-1 kerosene is a complex mixture of hydrocarbons with a chain length in the range of C₈–C₁₅ with a weighted average centered on C₁₁. The fractions obtained by rectification have lower boiling point and therefore present a lower carbon chain length: the weighted average is C₈–C₉, C₉–C₁₀ and C₁₀ for the 5%, 14% and 32% fractions, respectively. Besides the lower average carbon chain length, the most obvious effect of rectification is the reduction of sulfur content in the fractions: from the 228 ppm of the original Jet A-1 to 4 ppm in the 5% fraction, 12 ppm in the 14% fraction and 24 ppm in the 32% fraction. Another noticeable change is in the concentration of the cyclic hydrocarbons, aromatics and paraffins where, going toward lighter fractions the paraffins and aromatics content both decrease while the cyclic hydrocarbon content increases. This increased concentration in cyclic hydrocarbons, and the lower sulfur content are expected to have a beneficial effect on the PDh reaction. It has been observed in previous investigations that cyclic hydrocarbons are more easily dehydrogenated and, of all the compound classes, they give the major contribution to the hydrogen productivity [12,13].

3.2. Catalyst characterization

The catalyst used for the reaction of partial dehydrogenation of the kerosene fractions has the structure of a mesoporous alumina of rather low surface acidity. The X-ray diffraction pattern is dominated by the phases δ and γ Al₂O₃, while no diffraction peaks of BaO nor of tin or platinum oxides or metals are observed. The active phase is 1% Pt–1% Sn, and the metal dispersion is 30% (metal surface area 74 m² g^{−1}_{met}). The specific surface area is 100 m² g^{−1} with an average pore diameter of 18 nm [19].

3.3. Partial dehydrogenation

Each PDh reaction was carried out twice on each of the kerosene fractions and on the original Jet A-1 fuel reference, at 1 MPa, 450 °C, $\tau = 2$ s for a duration of 6 h. Good reproducibility of the hydrogen productivity and purity were found in each case, and the average productivity plots are shown in Fig. 4. The hydrogen productivity from all three kerosene fractions is improved with respect to that from Jet A-1, with the 5 wt.% fraction displaying the highest hydrogen production rate. PDh carried out on kerosene Jet A-1 (228 ppm S) show an immediate rapid drop in productivity due to catalyst deactivation. A similar effect is observed for the 32 wt.% fraction (24 ppm S), however the deactivation is slower and the hydrogen productivity stabilizes at higher values. A noticeable jump in productivity is observed between the 32 wt.% fraction and the 14 wt.% fraction (12 ppm S). The 5 wt.% (4 ppm S) and 14 wt.% fractions display a very different behavior with a remarkably higher stability and hydrogen productivity. This observation suggests that the different results are due both to the lower sulfur content and the change of chemical composition: the more marked drop in sulfur content occurs between Jet A-1 and the 32 wt.% fraction (from

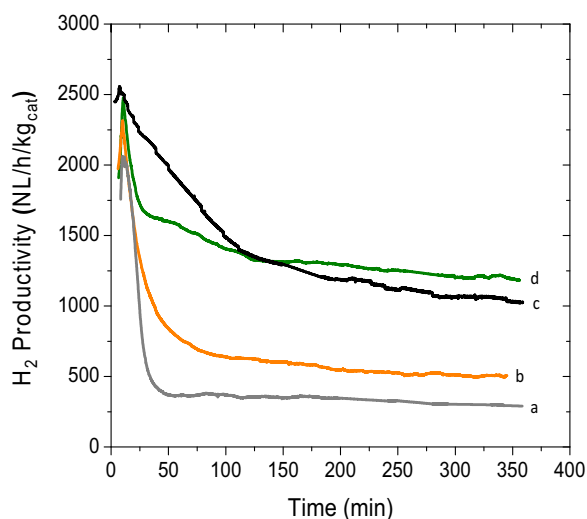


Fig. 5. Hydrogen productivity from PDh using rectified fractions and native Jet A-1 (a = Jet A-1; b = 32% fraction; c = 14% fraction; d = 5% fraction).

228 ppm to 24 ppm), but the greatest increase in stability and productivity are obtained with the 5 and 14 wt.% fractions. In order to better understand this behavior, the compositions of the fractions after reaction were compared to those of the freshly prepared fractions, Fig. 5. First, it is observed that the quantity of paraffins and iso-paraffins does not change during the reaction. Most importantly, it is seen that the cyclic hydrocarbons are the most reactive, these undergoing dehydrogenation to form aromatics, since the reduction in cyclic hydrocarbons is equal to the increase in aromatics content. Furthermore, it is observed that the amount of sulfur containing compounds is strongly decreased in the spent fractions after the PDh reaction. The variations in composition for each class of compound between the fresh and spent fractions are summarized in Table 1.

3.4. Catalyst deactivation

Sulfur is a well-known poison for platinum catalysis so there is the possibility that the sulfur contained in the kerosene is retained on the catalyst active phase by formation of sulfides during reaction [22,23]. Elemental analysis performed on the spent catalysts shows the presence of sulfur, but in a quantity significantly lower than the sulfur initially present in the respective fraction used for the reaction. This suggests that sulfur could perhaps have been

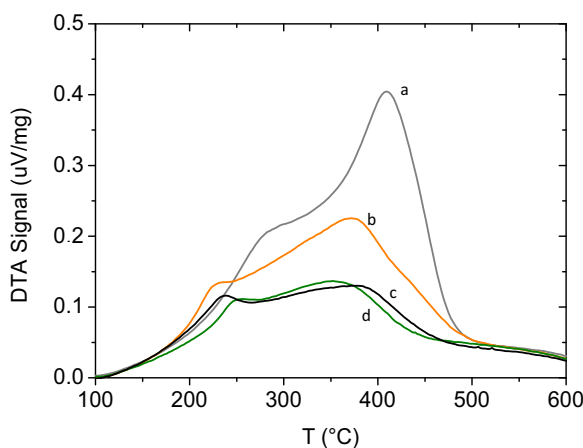


Fig. 6. DTA signal of the used catalysts, following PDh reaction (a = Jet A-1; b = 32% fraction; c = 14% fraction; d = 5% fraction).

Table 1
Modification of the composition of the liquid stream following PDh of rectified kerosene fractions.

Fraction	Iso-Paraffins	Cyclic	Dicyclic	Aromatics	Diaromatics	n-Paraffins	Sum
5%	−0.29	−4.88	0.04	6.10	0.00	−0.98	−0.01
14%	0.29	−4.58	0.09	4.72	0.00	−0.52	0.00
32%	0.44	0.39	0.16	−0.30	0.03	−0.71	0.01

removed during the reaction in a gaseous form, possibly as H_2S . Further studies will have to be carried out to establish the fate of the sulfur removed from the rectified streams during PDh. The results of GC analysis of the gaseous products produced during PDh is shown in Table 2. Hydrogen purity is in general very high and in all cases close to 99%, even with Jet A-1, with the highest purity being observed for the 5% fraction and the lowest for the standard Jet A-1. This effect could be due both to the different amount of sulfur in the liquid and the different chemical composition of the fractionated/original kerosene feeds. One possible explanation for the lower purity with fractions higher in sulfur is the formation of platinum sulfides in the course of the PDh reaction. The presence of PtS is known to decrease selectivity toward dehydrogenation and hydrogenolysis reactions [24–26]. This would be in agreement with the decrease in hydrogen production and the proportional increase of CH_4 (produced by cracking reactions) observed when using as reagent fractions with higher sulfur content. Overall the hydrogen purity is higher for the kerosene fractions produced by the rectification process. The spent catalysts were analysed by TGA and elemental analysis for the amount of residual carbon (coke) and the results are reported in Table 3. Good agreement is obtained for each of the samples using these two techniques. The quantity of carbon coke increases with the fraction weight in the order 5% fraction <14% fraction <32% fraction <Jet A-1 and, predictably, the amount of carbon follows the rate of catalyst deactivation. As reported in Fig. 6 the TG/DT analyses show that the carbon combustion peak comprises two contributions: a first peak at lower temperature (200–250 °C) and a second at higher temperatures (350–450 °C). From previous studies, it is assumed that the first peak is related to coke formed on the active metal phase and the second peak to coke formed on acid sites of the alumina support. The first type of carbon burns at lower temperature because its oxidation is promoted by the proximity of Pt particles and it is the major factor for catalyst deactivation as this carbon “covers” the active phase. The second type of coke is in strong interaction with the support and burns at a higher temperature; it affects the deactivation much less because it does not interact directly with the active phase [21,25]. The peaks areas reflect the deactivation trend observed during the PDh reaction. For the reference Jet A-1 feedstock, for which quickest deactivation was observed, the area of the peaks is remarkably higher than for the catalysts used in PDh of fractionated kerosene. The area of the first peak seems directly related to the deactivation ratio (defined as ratio between initial and final H_2 productivity), while the second peak area has been attributed to a type of coke formed in proximity to the support acid sites via polycondensation of aromatics formed during reaction. This type of coke could also contribute to catalyst deactivation via pore plugging [27–29]. The amount of sulfur on the spent catalysts measured by elemental analysis follows the trend of the amount of sulfur present in the fraction used as reagent and is also related to the catalyst

Table 2
Composition of the gas stream produced by PDh of rectified kerosene fractions.

	H_2 (%)	CH_4 (%)	C_2-C_4 (%)
5% Fraction	99.0	0.9	0.1
14% Fraction	98.8	1.1	0.1
32% Fraction	98.6	1.2	0.2
Jet A-1	98.6	1.1	0.3

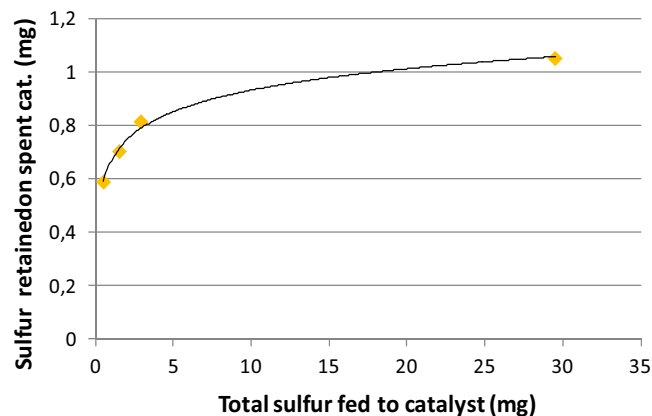


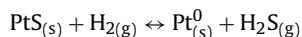
Fig. 7. Dependence of the amount of sulfur (in sulfur-containing molecules) retained by the catalyst on the total amount of sulfur fed to the catalyst.

performance. The deactivation in this case is caused by the two effects of carbon deposition and sulfur poisoning; the sulfur poisoning depends on the amount of sulfur present in the fraction, while the carbon deposition depends both on the fraction chemical composition and the effect of sulfide formation on the active phase. Using the results of elemental analysis, it is possible to calculate exactly how much sulfur is deposited on the spent catalyst and compare it to the total amount of sulfur flowed through the reactor (from the sulfur content in the pumped fuel). The amount of the sulfur deposit on the spent catalysts is proportional to the amount contained in the relative fraction used as reagent; however, except for the reaction with the 5 wt.% fraction, not all the sulfur introduced in the system is retained on the catalyst. Comparison between the sulfur retained on the catalyst as function of the sulfur content in the liquid fraction before reaction is shown in Fig. 7. It is known that, under the appropriate operative conditions, Pt/ γ - Al_2O_3 is an efficient deep hydrodesulfurization (HDS) catalyst and that even the sulfide PtS is active in the HDS reaction, presenting similar or higher activity as the well-known catalytic systems based on CoMo sulfides and NiMo sulfides [30]. The fact that the sulfur contained in the kerosene fractions is completely eliminated in the course of reaction provides proof that an HDS reaction takes place in PDh reaction conditions as well. The amount of sulfur retained on the spent catalysts can be attributed in minor part to sulfur containing hydrocarbons that remain on the pores after the reaction, but the majority of the sulfur probably originates from the PtS and SnS_2 formed during reaction [27,31]. The sulfidation of Pt is a partially reversible process: with the exception of a small amount of S that is irreversibly absorbed by Pt causing poisoning, PtS is instable

Table 3
Carbon coke analysis on spent catalysts after PDh of kerosene fractions.

	Elemental Analysis			TGA
	C %	H %	S %	Coke %
5% Fraction	0.98	0.36	0.064	1.1
14% Fraction	1.06	0.38	0.07	1.1
32% Fraction	1.73	0.37	0.093	1.9
Jet A-1	2.94	0.44	0.104	3.4

under hydrogen pressure and the amount formed depends on the concentration of S and H₂ in the reaction atmosphere [30]:



During the PDh reaction here, the Pt-Sn catalyst activates both the dehydrogenation and the hydrodesulfurization reactions and the sulfur contained in the liquid fraction is released in the form of H₂S, moving the equilibrium of the former equation to the left. The amount of sulfur retained in the catalyst is therefore proportional to the quantity of sulfur contained in the liquid. No H₂S was detected by GC analysis even though a small reduction of the hydrogen purity was measured with the increase of the sulfur content in the fraction. Assuming that all the sulfur not retained in the catalyst is released in form of H₂S, the volumetric % of H₂S in the hydrogen outflow has been calculated. This value is zero for the 5 wt.% fraction because all the sulfur contained in the liquid is retained on the spent catalyst, while for the 14 wt.% and 32 wt.% fractions the H₂S concentration in the produced gas is 0.007 and 0.026 vol%, respectively, which is below the detection limit of the GC. The presence of sulfur in the outflow though, even if very low, could poison the fuel cell catalyst downstream of the PDh reactor, and in order to utilize this system for their alimentations a sulfur trap needs to be placed after the reactor to optimize the total efficiency and lifetime of the system. However fractionation of kerosene Jet A-1 has been definitively identified by this study to be a promising processing step for the enhancement of hydrogen generation via partial dehydrogenation reaction, with an increase of the hydrogen productivity and purity and catalyst projected lifetime observed for all the fractions tested.

4. Conclusions

The fractionation of kerosene Jet A-1 by rectification is a novel approach for the enhancement of hydrogen generation via partial dehydrogenation, with an increase of hydrogen productivity and catalyst stability observed for all the fractions examined. The use of a 5 wt.% fraction increases the net hydrogen productivity threefold with respect to Jet-A1 reference test. The stability is also increased giving a value of more than 1000 NL · h⁻¹ · kg_{cat}⁻¹ after 6 h of reaction. The deactivation process involves both compositional changes in fractions and sulfur content in the reactor feed. The presence of a high proportion of cyclic hydrocarbons is beneficial for the hydrogen productivity because this class of compound is easily dehydrogenated with high selectivity to the corresponding aromatics, resulting in a high amount of hydrogen produced. Sulfur poisoning causes loss of hydrogen production with time, but for light fractions, such as 5 wt.%, almost the totality of sulfur-containing compounds are eliminated during the rectification process. This fuel treatment technique would allow the use of standard Jet A-1 kerosene instead of a low sulfur kerosene as feedstock for the partial dehydrogenation reaction,

reducing the problem of sulfur poisoning of the catalyst for the PDh reaction.

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